TEST METHOD

RESTRICTED

SUBJECT	SEQUENCE NUMBER
DETERMINATION OF AREA PERCENT PURITY OF EASTMAN™ PRODUCTS, CHDM-D, VERITYL AB-1000, CHDM-D90 GLYCOLS AND HIGH-BOILING CHDM DISTILLATES BY GAS CHROMATOGRAPHY	**EFFECTIVE DATE** September 22, 2011
	PAGE 1 **OF** 6 **PAGES**
	ACCESSION NUMBER
	ECCN EAR99
METHOD NO. EMN-A-AC-G-GC-500-7-SPEC	**SUPERSEDES ED.** 6 **DATED** 10/28/2009

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1. SCOPE AND SIGNIFICANCE

This method describes a gas chromatographic procedure for determination of the area percent purity of 1,4-cyclohexanedimethanol (CHDM) and reporting the percent of the major impurities present in CHDM-D, Verityl AB-1000, and CHDM-D90 glycols. This method is applicable to all CHDM samples from the refining columns. The range of this method on any of the impurities is from 0.05 to 50 percent, and for the CHDM is 0.1 to 99.9 percent. The analyst time is ten minutes, and the instrument time is twenty-five minutes.

2. SUMMARY OF METHOD

The sample is dissolved in dimethylformamide (DMF) and chromatographed on a (50%-phenyl)-methylpolysiloxane capillary column using a flame ionization detector. The concentrations of sample components are calculated from the integrated chromatogram using area normalization.

3. DEFINITIONS

The terms employed in this method are commonly used in normal laboratory practice and require no special comment.

4. PRECISION AND BIAS

4.1 Precision

The relative standard deviations are based on thirty injections of one prepared standard. Relative standard deviations ranged from 0.31% to 1.13% for individual components.

4.2 Bias

No calibration standards are used for area percent determinations; therefore, the accuracy has not been determined.

EASTMAN

Eastman Chemical Company
Kingsport, Tennessee 37662

5. SAFETY PRECAUTIONS

5.1 Nitrile gloves must be worn when handling DMF. Work should be performed in a well-ventilated area to minimize vapor inhalation. If exposure to the vapors cannot be avoided (for example, if the sample must be heated without a cover or volumes greater than 20 mL must be transferred), the work must be performed in a hood.

5.2 Otherwise, normal safety precautions and safe handling practices should be observed.

5.3 Refer to the PPE Matrix posted in the lab for required personal protective equipment.

6. SOURCES OF ERROR

The method is an area percent determination where each component is given the same detector response factor of 1.0000, and the total data is normalized to 100%. If the assumption of equal detector response factors for every component is incorrect, the area percent cannot be taken to represent the weight percent of each component in the solution. Also, the method will not account for non-eluting components.

7. APPARATUS

7.1 Gas chromatograph with split injector and FID

7.2 Autosampler

7.3 Chromatography data system

7.4 Vials, autosampler with septum caps

7.5 Capillary column, fused-silica, (50%-phenyl)-methylpolysiloxane, 30-m x 0.32-mm id with film thickness = 0.25 µm

7.6 Disposable transfer pipets

7.7 Vials, 8-dram

7.8 Karl Fischer titrator

8. REAGENTS AND MATERIALS

Dimethylformamide (DMF) (R-3, S-3, F-2, C-0)

9. CALIBRATION AND STANDARDIZATION

This method uses an area percent calculation because of the difficulty in obtaining pure materials. If possible, a sample of crude CHDM containing high levels of impurities should be obtained and used to verify the retention times.

10. SAMPLE PREPARATION

10.1 Ten grams of the colorless sample are required for a single analysis.

10.2 If the sample as received has two layers, shake vigorously before taking a sample. If the sample has solidified, warm it up in an oven at approximately 100°C until it is a liquid.

11. PROCEDURE

11.1 If the sample label asks for percent water by Karl Fischer titration, the GC results are corrected for water automatically in LIMS.

11.2 Transfer approximately 0.2 grams of sample into an 8-dram vial.

11.3 Add 5 mL of dimethylformamide (DMF) to the vial, cap and shake well to dissolve.

11.4 Transfer approximately 1 mL of the sample prepared in Step 11.3 to an autosampler vial and cap the vial.

11.5 Record and integrate the chromatogram using the conditions specified in Section 7 and Appendix A1.

12. CALCULATIONS

All calculations are performed by the chromatography data system, or the area percents of each component can be calculated by hand as follows:

$$\text{Area \% Component} = \frac{(\text{Area of Component}) \times 100}{\text{Sum of Areas of All Detected Components Excluding DMF Solvent}}$$

$$\text{Area \% Component (corrected for water)} = \frac{(\text{Area of Component}) \times (100 - \%\text{Water})}{\text{Sum of Areas of All Detected Components Excluding DMF Solvent}}$$

ppm = Area% x 10,000

13. REPORT

Report the percents of the identified components to one decimal place.

14. BIBLIOGRAPHY

None

15. APPENDIX

A1. Instrument Conditions

INSTRUMENT CONDITIONS
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Oven Parameters
Oven: On
Oven Equilib Time: 0.50 min
Oven Max. Temperature: 320 C
Oven Cyro: Off
Oven Cyro Blast: Off
Oven Ambient: 20 C
Timeout Detection: Off
Timeout Detector: 10.00 min
Fault Detection: Off

Oven Temperature Program

Level	Rate	Next Temp	Hold Time
Initial		115.00 C	0.00 min
1	5C/min	130.00 C	0.00 min
2	100 C/min	200.00 C	0.00 min
3	120 C/min	280.00 C	3.00 min

Zone Temperatures
Front Inlet Setpoint: 280 C
Back Inlet Setpoint: 280 C
Front Detector Setpoint: 280 C
Back Detector Setpoint: 280 C

Inlet Temperature Setup

Inlet: Front
Oven Track: Off
Zone Temperature: 280 C
Inlet Temperature Program: Off

Inlet: Back
Oven Track: Off
Zone Temperature: 280 C
Inlet Temperature Program: Off

Inlet Pressure Setup

Inlet: Front
General Inlet Settings
S/SL, COC, PP Inlet
Split Mode: Split
Split Flow: 269 mL/min
Split Ratio: 75.00 : 1
Gas Saver: 20.00 mL/min @ 2.00 min

INSTRUMENT CONDITIONS
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Inlet: Back
General Inlet Settings
S/SL, COC, PP Inlet

Split Mode:	Split
Split Flow:	269 mL/min
Split Ratio:	75.00 : 1
Gas Saver:	20.00 mL/min @ 2.00 min

Column Setup

Column 1
Capillary Column

Length:	30.00 m
Inside Diameter:	0.25 mm
Film Thickness:	0.25 um
Inlet:	Front
Detector:	Front
Gas:	Hydrogen

Carrier Flow

Column Mode:	Constant Flow
Flow:	3.50 mL/min

Column 2
Capillary Column

Length:	30.00 m
Inside Diameter:	0.25 mm
Film Thickness:	0.25 um
Inlet:	Back
Detector:	Back
Gas:	Hydrogen

Carrier Flow

Column Mode:	Constant Flow
Flow:	3.50 mL/min

Signals Setup

Signal 1 Parameters

Detector:	Front Det
Range:	0
Attenuation:	0
Auto Zero:	Off

Signal 2 Parameters

Detector:	Back Det
Range:	0
Attenuation:	0
Auto Zero:	Off

INSTRUMENT CONDITIONS
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Run Time Control Setup

Run Time Control Program

#	Time (min)	Event	Parameter	Value

Front FID Setup

Heater:	280 C
H2 Flow:	30 mL/min
Air Flow:	350 mL/min
Makeup Flow:	
Makeup Flow Type:	Helium
Constant Column:	30 mL/min
Flame:	On
Electrometer:	On

Back FID Setup

Heater:	280 C
H2 Flow:	30 mL/min
Air Flow:	350 mL/min
Makeup Flow:	
Makeup Flow Type:	Helium
Constant Column:	30 mL/min
Flame:	On
Electrometer:	On

Autosampler Instrument Setup

Front Injector Program

Sample Washes:	4
Sample Pumps:	4
Viscosity Delay:	0
Solvent A Post-washes:	4
Solvent B Post-washes:	4

Rear Injector Program

Sample Washes:	4
Sample Pumps:	4
Viscosity Delay:	0
Solvent A Post-washes:	4
Solvent B Post-washes:	4

Trigger Type: External Trigger